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(54) METHOD FOR MANUFACTURING BASE MATERIAL FOR OPTICAL FIBER

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(72) Inventor: Hiroshi Yokoda
Yokohama Works, Sumitomo Electric Industries, Ltd.
1 Taya-cho, Sakae-ku, Yokohama-shi, Kanagawa-ken
(72) Inventor: Toshio Dantsuka
Yokohama Works, Sumitomo Electric Industries, Ltd.
1 Taya-cho, Sakae-ku, Yokohama-shi, Kanagawa-ken
(72) Inventor: Sanezumi Ito
Yokohama Works, Sumitomo Electric Industries, Ltd.
1 Taya-cho, Sakae-ku, Yokohama-shi, Kanagawa-ken
(72) Inventor: Masahiro Takashiro
Yokohama Works, Sumitomo Electric Industries, Ltd.
1 Taya-cho, Sakae-ku, Yokohama-shi, Kanagawa-ken
(72) Inventor: Hiroo Kanemori
Yokohama Works, Sumitomo Electric Industries, Ltd.
1 Taya-cho, Sakae-ku, Yokohama-shi, Kanagawa-ken
(71) Applicant: Sumitomo Electric Industries, Ltd.
5-15 Kitahama, Higashi-ku, Osaka-shi, Osaka-fu
(74) Agents: Akira Uchida, patent attorney, and 3 others

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CLAIMS

1. A method for manufacturing a base material for an optical fiber, characterized by the fact that in a method for manufacturing a base material for an optical fiber by heating a porous base material having a core-corresponding part and a clad-corresponding part and making it transpartent, it forms a porous base material for the core by depositing fine glass particles generated by supplying a glass raw material into a burner flame by subjecting it to a gas-phase

reaction, transforms it into a porous base material for a core with a raised bulk density by heating said porous base material for the core, and obtains a composite porous base material by depositing fine glass particles for the clad at the outer periphery of said porous base material.

2. The method for manufacturing a base material for an optical fiber of Claim 1, characterized by the fact that the heating is carried out in a range in which the average bulk density $\overline{\rho}$ (g/cm$^3$) of the porous base material for the core is $0.4 \leq \overline{\rho} \leq 1.0$.

## DETAILED EXPLANATION OF THE INVENTION

### INDUSTRIAL APPLICATION FIELD

The present invention pertains to the improvement of a method for manufacturing a base material for an optical fiber by a gas-phase synthesis method.

### PRIOR ART

As a method for manufacturing a base material for an optical fiber by a gas-phase synthesis method, a conventional VAD (Vapor-Phase Axial Deposition) method is known. In the method, as shown in Figure 1, a raw material gas for forming a glass is supplied with a combustion gas such as $H_2$ and $O_2$ and an inert gas such as Ar to a burner 1 for synthesizing glass fine particles (hereinafter, abbreviated as a burner). As the glass raw material, $SiCl_4$ is usually used, and an additive raw material such as $GeCl_4$ and $SiF_4$ may also be mixed to adjust the refractive index. As the combustion gas, for example, a hydrocarbon-containing gas such as $CH_4$, $C_2H_6$, and $C_3H_8$, CO, etc., can also be used. Said raw material gas for forming a glass is flame-hydrolyzed in a flame (oxyhydrogen flame in case $H_2$ and $O_2$ are used) of the burner 1, so that fine glass particles are generated. The fine glass particles begin to be attached from the tip of a starting rod 2 installed in a rotary lifter 4 and lifted while rotating said starting rod 2, so that a porous base material 3 is formed in the axial direction of the starting rod 2. The porous base material 3 manufactured in this manner is subjected to a heating and dehydrating treatment (hereinafter, called a heating-dehydrating treatment) and a heating and transparency-promoting treatment (hereinafter, called a heating-dehydrating treatment), so that a transparent glass base material is obtained. Said transparent glass base material is transformed into a stretched rod for the core by stretching at a prescribed diameter, and said rod for the core is mounted as a starting material 5 in the rotary lifter 4 in the apparatus constitution as shown in Figure 2. Using a burner 6 for synthesizing the clad, the fine glass particles are deposited at the outer periphery of said rod for the core, so that a composite body composed of the rod for the core and the porous body for the clad is formed. A base material for an optical fiber with the desired clad/core diameter ratio

is obtained by subjecting said composite body to the heating-dehydrating treatment and the heating-transparency treatment similarly to the above case.

## PROBLEMS TO BE SOLVED BY THE INVENTION

On the other hand, in the above-mentioned conventional method, the transparent glass base material for the core is transformed into the rod for the core by stretching at a prescribed diameter; however, a flame polishing is required to clean and smooth the outer surface of said rod before depositing the fine glass particles for the clad at the outer periphery of said rod. Usually, an oxyhydrogen flame is used in the flame polishing; however, since the surface of the rod for the core is exposed to the flame, high-concentration OH groups are mixed into the core surface. Furthermore, in the deposition of the fine glass particles for a clad in the subsequent process, since the outer surface of the rod for the core is also heated by the flame of the burner, the OH groups are also mixed into the core. Since the existence of the OH groups markedly degraded the transmission loss, which was an important characteristic of optical fibers, the OH group mixture was a very difficult problem.

The purpose of the present invention is to offer a method that solves the problem of the OH group mixing into the core, and that can manufacture a base material for a high-quality optical fiber with excellent transmission characteristics; the method forms fine glass particles from such a glass raw material by the gas-phase synthesis method, obtains a porous base material by depositing them, and obtains an optical fiber base material by transforming it into a transparent glass.

## MEANS TO SOLVE THE PROBLEMS AND OPERATION

The present invention pertains to a method for manufacturing a base material for an optical fiber, characterized by the fact that in a method for manufacturing a base material for an optical fiber by heating a porous base material having a core-corresponding part and a clad-corresponding part, and makes it transparent, it forms a porous base material for a core by depositing fine glass particles generated by supplying a glass raw material into a burner flame by subjecting it to a gas-phase reaction, transforms it into a porous base material for the core with a raised bulk density by heating said porous base material for the core, and obtains a composite porous base material by depositing fine glass particles for the clad at the outer periphery of said porous base material.

As an especially preferred embodiment of the present invention, the heating is carried out in a range in which the average bulk density $\overline{\rho}$ (g/cm$^3$) of the porous base material for the core is $0.4 \leq \overline{\rho} \leq 1.0$.

In the present invention, the heating-dehydrating treatment after forming the porous base material for the core is similar to the conventional method; however, said dehydrated porous body is heated and shrunk, so that its average bulk density is raised. More preferably, the porous body is heated and shrunk so that the average bulk density $\overline{\rho}$ may be $0.4 \leq \overline{\rho} \leq 1.0$. Such a heating is carried out by maintaining the temperature at 1,200-1,500°C in a He gas atmosphere using an ordinary electric furnace.

In this heated and shrunk state, that is, without a transparency-promoting treatment, stretching, and flame-polishing, the porous base material for the core is mounted as a starting material 5 in a lift rotator 4 with the constitution shown in Figure 2, then fine glass particles for the clad are deposited at its outer periphery by a well-known means using a burner 6 for the clad and grown in the axial direction, so that a composite porous base material composed of the above-mentioned porous base material for the core (starting material 5) and a porous body 7 for the clad is obtained. A transparent glass base material having a core and a clad is obtained by subjecting said composite porous base material to the heating-dehydrating treatment and the heating-transparency-promoting treatment, and it is wire-drawn, so that an optical fiber is obtained.

OPERATION

In the present invention, since the porous base material for the core is not subjected to the transparency-promoting treatment, stretching, and flame-polishing treatment, there is no mixture of OH groups due to the stretching and the flame polishing. Therefore, unlike the conventional method, a high-concentration OH group layer is not formed at the core and clad interface.

Also, as for the porous base material for the core, which is heated and shrunk so that the average bulk density $\overline{\rho}$ may be raised, preferably so that $\overline{\rho}$ is $0.4 \leq \overline{\rho} \leq 1.0$, when fine glass particles for the clad are deposited on it in the next process, since the porous base material is heated by a flame of the burner, OH groups are introduced into the outer periphery. However, after forming the porous body for the clad, the OH groups introduced into the above-mentioned OH groups in the above-mentioned core are removed at the time of the heating-dehydrating treatment of the composite porous base material. At that time, if the average bulk density $\overline{\rho}$ of the porous base material for the core is greater than 1, the OH group removal from the outer surface of the porous base material for the core is insufficient, which is not preferable. Also, if the $\overline{\rho}$ is less than 0.4, cracks are easily generated in the vicinity of the interface with the porous base material for the core when the fine glass particles for the clad are deposited, so that a stable manufacture is difficult, which is not preferable. The average bulk density of the porous body prepared by the ordinary VAD method, etc., is about 0.2-0.3.

According to the present invention, several porous base materials for a core of the clad type fiber were heated and shrunk so that the average bulk density $\overline{\rho}$ had different values, so that a composite porous base material having a core and a clad was obtained. An optical fiber was then obtained by wire-drawing after the transparency treatment, and its OH absorption-loss increase at a wavelength of 1.38 μm was investigated. As for the results, the average bulk density $\overline{\rho}$ ($g/cm^3$) of the porous base material for the core after heating and shrinking is indicated on the abscissa, and the OH absorption-loss increase (dB/km) of the fiber obtained is indicated on the ordinate. The relationship between them is shown as a graph in Figure 3.

The manufacture of the porous base material by depositing the fine glass particles synthesized in the gas phase from the glass raw material may be carried out by a conventional well-known technique; for example, the VAD method, OVFD method, plasma flame method, etc., can be mentioned.

As the glass raw material for a core or clad, for example, $SiCl_4$, $SiHCl_2$, etc., can be used, and as additives for adjusting the refractive index, etc., for example, $GeCl_4$, $SiF_4$, $SF_6$, $CCl_2F_5$, etc., can be used.

As a fuel gas and a combustion gas, in general $H_2$, $CH_4$, $C_2H_6$, $O_2$, CO, etc., can be used, and as an inert gas, Ar, $N_2$, etc., can be used.

The heating-dehydrating treatment of the porous base material for the core is carried out by heating at a temperature of 800-1,100°C in a mixed atmosphere of He gas and a $Cl_2$ or Cl compound gas using an electric furnace, for instance.

The porous body for a core that is heated and dehydrated is heated at a temperature of 1,200-1,500°C in a He gas atmosphere using the electric furnace as mentioned above, so that the bulk density is raised.

Also, the process for forming the composite porous base material by depositing the fine glass particles for a clad at the outer periphery of the porous base material for the core, of which the bulk density is raised, may be carried out similarly to the formation of the above-mentioned porous body for a core, and the heating-dehydrating treatment and the heating-transparency-promoting treatment of said porous base material may also be similarly carried out.

## APPLICATION EXAMPLES

### APPLICATION EXAMPLE 1

In the apparatus constitution shown in Figure 1, $H_2$ at 2.35 L/min, $O_2$ at 7 L/min, Ar at 4 L/min, $SiCl_4$ at 560 cc/min, and $GeCl_4$ at 32 cc/min were supplied to a burner for the core, and a porous body for the core was manufactured at a lift rate [in the rotator] of 59 mm/min. The

outer diameter of the porous body for the core obtained was 87 mm $\phi$, and the average bulk density was 0.19 g/cm$^3$. Said porous body was heated and dehydrated at a temperature of 1,050°C in a He gas atmosphere containing 6 vol% $Cl_2$ (gas partial pressure ratio) using an electric furnace. Next, it was heated at 1,400°C in an atmosphere of only He gas, so that the average bulk density of said porous base material was controlled to 0.72.

Using the porous base material for a core heated and shrunk as a starting material 5 in the apparatus constitution shown in Figure 2, a porous body for the clad was formed at its outer periphery. At that time, $H_2$ at 38 L/min, $O_2$ at 45 l/min, Ar at 13 L/min, and $SiCl_4$ at 3.20 L/min were supplied to a burner for the clad, and the lift rate was set to 84 mm/min. The bulk density of the porous body part for a clad was 0.31 g/cm$^3$.

The composite porous base material obtained was heated and dehydrated at a temperature of 1,060°C in a He gas atmosphere containing 6 vol% $Cl_2$ using the electric furnace. Next, it was heated at 1,620°C in an atmosphere of only He gas, so that a transparent glass was obtained. At the core and clad interface of the transparent glass parent material obtained, the generation of bubbles was not seen.

A clad fiber with an outer diameter of 125 µm and a refractive index difference of 0.94% was prepared by wire-drawing said transparent glass base material; when the optical transmission loss was measured, favorable characteristics, i.e., an OH absorption loss of 0.31 dB/km at a wavelength of 1.38 µm and 0.52 dB/km at a wavelength of 1.3 µm were shown.

COMPARATIVE EXAMPLE 1

Similarly to Application Example 1 except for raising the temperature for heating and shrinking a porous base material for the core to 1,600°C, a transparent glass was obtained. Without stretching the rod, fine glass particles for the clad were deposited at its outer periphery under the same conditions as those of Application Example 1, similarly heated and dehydrated, and subjected to a transparent-glass formation treatment, so that a transparent glass base material in which bubbles were generated at part of the interface of the core and the clad was obtained. The part without bubbles of said base material was wire-drawn into a fiber; when its transmission loss was measured, the OH absorption loss was 3.0 dB/km at a wavelength of 1.38 µm and 1.2 dB/km at a wavelength of 1.3 µm. These values were higher than those of the product of the present invention of Application Example 1, and the transmission characteristics were inferior.

APPLICATION EXAMPLE 2

In the apparatus constitution shown in Figure 1, $H_2$ at 2.8 l/min, $O_2$ at 9 L/min, Ar at 5 l/min, and $SiCl_4$ at 350 cc/min were supplied to a burner for the core, and a porous body for the core was manufactured at a lift rate of 80 mm/min. The outer diameter of the porous body for the

core obtained was 55 mm $\phi$, and the average bulk density was 0.27. Said porous body was heated and dehydrated at a temperature of 1,050°C in an atmosphere of He at 10 L/min He and $Cl_2$ at 500 cc/min using an electric furnace. Next, it was heated and shrunk at 1,460°C in an atmosphere of only He at 10 L/min, so that the average bulk density was increased to 0.92.

As for said porous base material that was heated and shrunk, in the apparatus constitution shown in Figure 2, $H_2$ at 44 L/min, $O_2$ at 62 L/min, Ar at 15 L/min, and $SiCl_4$ at 7.4 L/min were supplied to a burner for the clad, and fine glass particles for the clad were deposited at a lift rate set to 71 mm/h, so that a composite porous body with an outer diameter of 210 mm $\phi$ was obtained. The composite porous base material was heated and dehydrated at a temperature of 1,040°C in a He gas atmosphere containing 6 vol% $Cl_2$ using the electric furnace. Next, it was heated at 1,250°C in a He gas atmosphere containing 2.7 vol% $SiF_4$, then F was added. This was heated at 1,610°C in a He gas atmosphere containing 2.7 vol% $SiF_4$, so that a transparent glass was formed.

After heating and stretching the glass rod obtained, fine glass particles for the clad were deposited again under conditions similar to the above-mentioned conditions, so that the clad/core diameter ratio was 15, and a transparent glass was obtained similarly to the above.

An optical fiber with an outer diameter of 125 μm was obtained by wire-drawing the base material rod for an optical fiber obtained in this manner. Said fiber was a single-mode fiber in which the refractive index difference of the $SiO_2$ clad added was 0.29%. It showed good characteristics, in which the cut-off wavelength $\lambda$ was 1.17 μm, the transmission loss was 0.34 dB/km at a wavelength of 1.3 μm and 0.19 dB/km at a wavelength of 1.55 μm, and the OH absorption loss was 0.9 dB/km at a wavelength of 1.38 μm.

## COMPARATIVE EXAMPLE 2

Similarly to Application Example 2 except for raising the temperature for heating and shrinking the porous base material for the core to 1,630°C, a transparent glass was obtained. Under the same conditions as those in Application Example 2, a base material rod for an optical fiber was obtained and drawn into a fiber. The OH absorption loss of said fiber was as large as 2.9 dB/km at a wavelength of 1.38 μm, and the transmission loss at a wavelength of 1.3 μm was 1.1 dB/km. These characteristics were considerably inferior to those of the product of the present invention of Application Example 2.

## EFFECT OF THE INVENTION

According to the present invention, without the heating, stretching, and flame-polishing of a porous body for a core resulting in the mixing of OH groups, said porous body is heated to raise the average bulk density, and a porous body for a clad is formed at its outer periphery.

Thus, the OH group mixing problem during the clad formation can also be solved.

Therefore, in the present invention, the OH absorption loss is reduced and this method is very favorable for manufacturing the base material for an optical fiber, which can manufacture an optical fiber with excellent transmission characteristics. Also, in the present invention, treatment processes of heating and transparency promotion, stretching, and flame-polishing of the porous body for the core can be omitted, which is industrially advantageous.

## BRIEF DESCRIPTION OF THE FIGURES

Figure 1 is an outlined cross section for explaining an embodiment of the processes for manufacturing a porous body for the core of the present invention.

Figure 2 is an outlined cross section for explaining an embodiment of the process for manufacturing a composite porous body by forming a porous body for a clad at the outer periphery of the porous body for the core of the present invention.

Figure 3 is a graph showing the relationship between the average bulk density ($g/cm^3$) of the porous base material for the core heated and shrunk and the OH absorption loss (dB/km) of a fiber obtained from said base material.

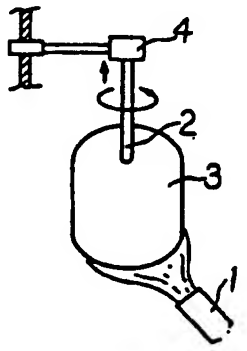


Figure 1

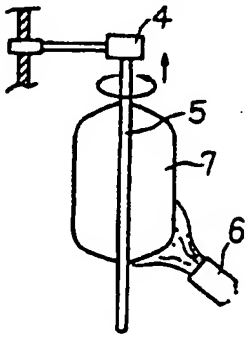


Figure 2

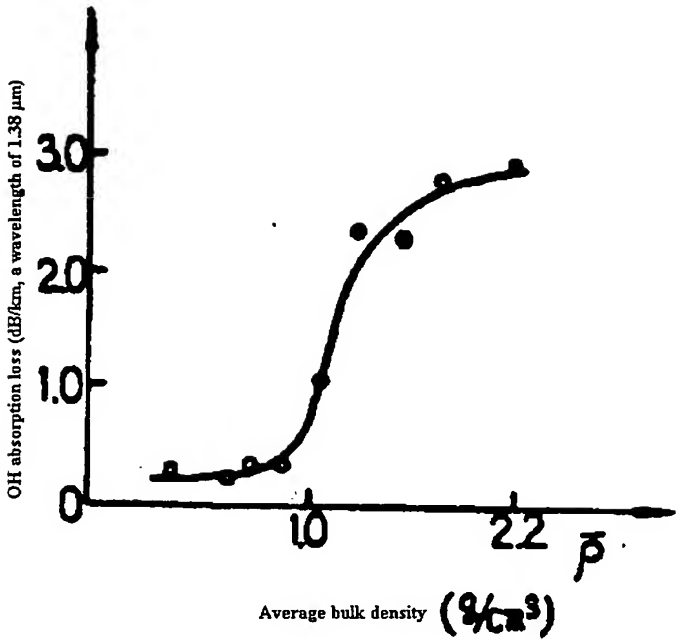
OH absorption loss (dB/km, a wavelength of 1.38 μm)
3.0
2.0
1.0
0
1.0
2.2
ρ̄
Average bulk density (g/cm³)

Figure 3

PHOENIX

TRANSLATIONS

*...the height of Excellence...*

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6306 HIGHLAND HILLS DR., AUSTIN, TX 78731 phone: (512) 343-8389,
toll-free 877-452-1348, fax (512) 343-6721, email: phoenixtranslations@ev1.net